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GELCAST AS800 MATERIALS CHARACTERIZATION FOR ROCKET ENGINE APPLICATIONS

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ABSTRACT

The material properties for a gelcast silicon nitride (AS800) manufactured by AlliedSignal Ceramic Components of Torrance, California were characterized under the Air Force sponsored Light Weight Thrust Chamber Assembly (LWTCA) Program. The testing results summarized in this paper include tensile, flexural strength, compressive strength, interrupted stress rupture, thermal cycle, fracture toughness, low cycle fatigue, and elastic moduli determinations. In addition, the results for a series of tests determining the effect of simulated rocket engine environment on AS800 are included. Specimens were exposed to hydrogen, water, and oxygen at approximate temperature and pressures of engine operation. After exposure, the retained flexural strength of the specimen was measured and compared to the strength of virgin material. Environmental effects for as-sintered specimens and machined specimen surfaces were measured. The test temperatures included cryogenic (-320°F), ambient and elevated (2000°F).

The characterization of AS800 yielded data consistent with AlliedSignal published data. The environmental exposure data showed that there are some effects on the retained properties of AS800 from exposure to oxygen and water combinations. The effects were statistically significant for as-sintered specimens exposed to oxygen with 10% water. The effects for other environmental conditions were not as severe and more data is needed to quantify the effects.

INTRODUCTION

The Light Weight Thrust Chamber Assembly (LWTCA) program is a 52 month Air Force program to develop a rocket engine following the IHPRPT (Integrated High Pay-Off Rocket Propulsion Technology) goals. The IHPRPT goals are to achieve 40% lower weight, 50% lower cost, a 75% reduction in the number of

parts, and 3% increase in I_{sp} over the benchmark engine design, the Space Shuttle Main Engine (SSME) Block 1 engine. Silicon nitride, due to its high specific strength, was the proposed advanced material baseline to be utilized on the major components of the chamber, including injector body and structural jacket.

Testing for complete materials characterization of the AS800 gelcast silicon nitride was performed by the Material Applications Department of Rocketdyne Propulsion & Power Division of the Boeing Company, Canoga Park, California, under LWTCA program funds. The objectives of the testing were threefold: 1) to verify AlliedSignal published data, 2) to obtain properties for elevated and cryogenic temperatures, and 3) to obtain material properties required for formulation of the design methodology. The tests summarized in this report were performed at four locations: University of Dayton Research Institute, Rockwell Science Center, Rocketdyne Environmental Effects Laboratory, and University of California at Los Angeles. In summary, all mechanical properties obtained by Rocketdyne for AS800 fall within the family of data supplied by AlliedSignal. Specimens that were in the original as-sintered surface finish exhibited a drop in the retained flexural strength.

Table I summarizes all the characterization tests including the conditions of testing. The tests are identified by test ID numbers. Gelcast AS800 test specimens were obtained from AlliedSignal Ceramic Components under LWTCA contract funds for all testing.

The majority of the mechanical testing was conducted at the Rockwell Science Center. Cryogenic testing at liquid nitrogen temperatures and high temperature testing at 2000°F were performed in addition to room temperature testing. The University of Dayton Research Institute (UDRI) performed the ORNL buttonhead testing. All buttonhead testing at UDRI was performed at cryogenic temperatures. The Rocketdyne Santa Susana Field Laboratory conducted the environmental exposures with hydrogen and oxygen. AS800 samples were exposed to the simulated conditions of LWTCA rocket engine operation. Pressure, temperature and water/fuel or water/oxidizer conditions of the engine were created in the laboratory.

CHARACTERIZATION TESTS

In Table I, the test procedures, raw data, and test results are organized by test ID numbers. Test ID numbers 1 through 5 consist of the mechanical baseline tests. These tests consisted of buttonhead and traditional dog-bone and 4-point bend test

Table I - AS800 Characterization Matrix

General Test Type	Test ID #	General Test Description or Environmental Exposure	Cryogenic (-300°F)	Ambient (70°F)	Elevated
Tensile	1	Fast Fracture	15		
	2	Fast Fracture#	7	12	12
4-Point Bend	3	Fast Fracture	12	8	12
	4	Fast Fracture *		12	
Compression	5	Fast Fracture#	2	4	2
Environmental Exposure (Retained Strength)	6	H_2 @ -320°F and 6,403 psi for 30 Minutes - Tensile Bar#		6	
	7	H_2 @ -320°F and 6,476 psi for 25 Hours - Tensile Bar#		6	
	8	H_2O @ 800°F and 1,035 psi for 30 Minutes		6	
	9	H_2O @ 800°F and 1,035 psi for 30 Minutes *		6	
	10	H_2O @ 800°F and 1,482 psi for 25 Hours		6	
	11	H_2O @ 800°F and 1,482 psi for 25 Hours *		6	
	12	H_2 with10wt% H_2O @ 800°F and 3,694 psi for 30 Minutes *		6	
	13	H_2 with10wt% H_2O @ 800°F and 3,550 psi for 25 Hours *		6	
	14	H_2 with10wt% H_2O @ 800°F and 3,790 psi for 30 Minutes		5	
	15	H_2 with10wt% H_2O @ 800°F and 3,635 psi for 25 Hours		6	
	16	He with 10wt% H_2O @ 800°F and 3,595 psi for 30 Minutes		6	
	17	He with 10wt% H_2O @ 800°F and 3,595 psi for 30 Minutes *		6	
	18	He with 10wt% H_2O @ 800°F and 3,587 psi for 25 Hours		6	
	19	He with 10wt% H_2O @ 800°F and 3,587 psi for 25 Hours *		6	
	20	O_2 with10wt% H_2O @ 800°F and 3,225 psi for 30 Minutes		6	
	21	O_2 with10wt% H_2O @ 800°F and 3,225 psi for 30 Minutes *		6	
	22	O_2 with10wt% H_2O @ 800°F and 3,438 psi for 25 Hours		6	
	23	O_2 with10wt% H_2O @ 800°F and 3,438 psi for 25 Hours *		6	
Interrupted Stress Rupture	24	110% Design Stress, 125% Design Life - Tensile Bar#	6	6	6
Thermal Cycle	25	50 Cycles from 1800°F to LN_2 Quench plus 50 Cycles from 2200°F to LN_2 Quench		4	
	26	100 Cycles from 2200°F to LN_2 Quench		6	
Fracture Toughness	27	~~Vickers indent followed by standard 4 pt bend~~	4	4	6
	28	Conventional fracture toughness test	3	1	2
Low Cycle Fatigue	29	300 cycles @ Max. Stress & Combined Thermal Cycle - Tensile Bar#		4	
Elastic Modulus/Poisson's	30	Test & data provided by the University of Dayton		6	

* Denotes an "As-Processed" specimen, always a flexural bar.
These specimens have one surface that is as cast and sintered, placed in tension for the retained strength tests.

\# Denotes an hour-glass tensile specimen

specimen geometries (figure 1). Two types of surface finish were tested on the specimens: "machined" and "as-processed".

The purpose of the testing on "machined" and "as-processed" specimens was to establish the effect of machining on AS800 characteristic mechanical properties. The specimens with machined surfaces were expected to exhibit higher mechanical properties than the as-processed specimens. The as-processed specimens were samples that had one surface left unmachined after sintering.

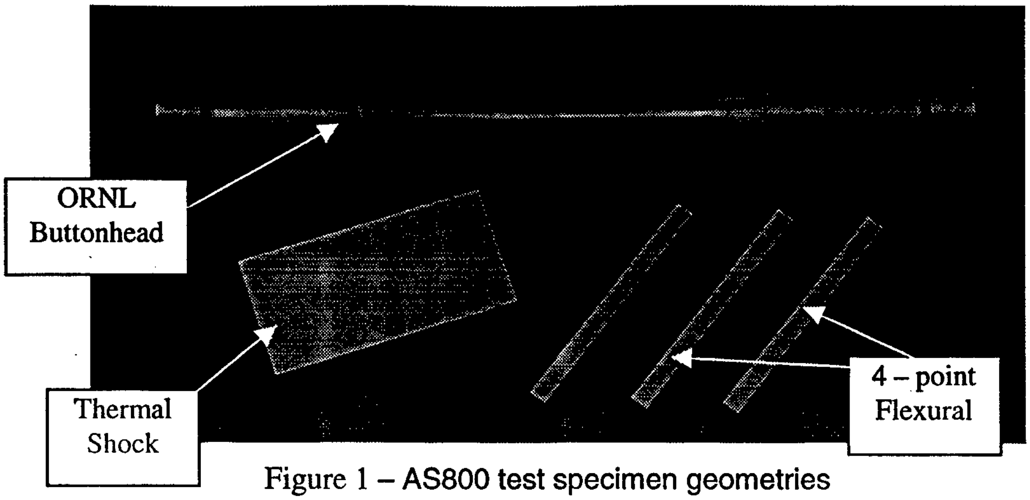


Figure 1 – AS800 test specimen geometries

The LWTCA engine hardware is expected to have both types of surfaces.

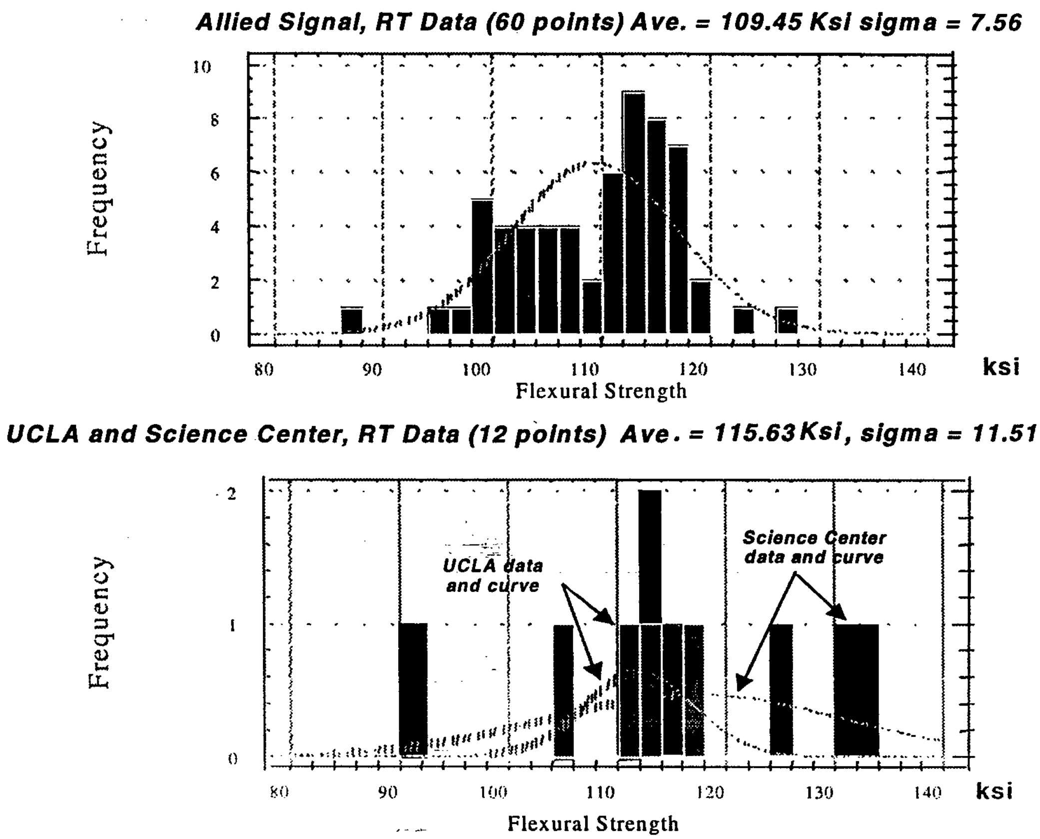
All the ORNL buttonhead specimens were tested at cryogenic (-320°F) temperature to allow for a statistically significant database. To date no cryogenic strength data has been published by AlliedSignal. The values from the mechanical testing are compared against the AlliedSignal data in figures 2-4.

In summary, the mean strength obtained by Rocketdyne for the flexural strength samples fell within the family of data generated by AlliedSignal. Wiebull moduli comparisons for the room temperature, cryogenic (-320°F) and high temperature (1832°F) are very close to the published numbers by AlliedSignal.

ENVIRONMENTAL TESTS

To simulate the conditions of engine operation for the LWTCA, the pressure, temperature and fuel/water or oxidizer/water conditions of the engine combustion products were simulated in a laboratory environment (test IDs 6-23). AS800 samples (machined and as-processed) were exposed to these conditions followed by mechanical testing. The test conditions are summarized below:

- H_2 at –320°F and 6400 psi, 30-minute and 24-hour durations
- H_2O at 800°F and 6400 psi, 30-minute and 24-hour durations
- H_2 with 10% water at 800°F & 3690 psi, 30-minute and 24-hour durations
- He with 10%water at 800°F & 3600 psi, 30-minute and 24-hour durations
- O_2 with 10%water at 800°F & 3600 psi, 30-minute and 24-hour durations



• Mean strength obtained by Rocketdyne in the family reported by AS

Figure 2 – AS800 Flexural Strength, Room Temperature Rocketdyne and AlliedSignal Data Comparison

Testing was performed by exposing samples to 1) 100% steam and 2)He with 10% water to establish baselines. Three environmental exposure chambers were machined for this testing. For test IDs 6-23, durations of 30-minute and 25-hour exposures were selected based on the maximum LWTCA engine operational cycle. After each exposure cycle the specimens were tested for retained flexural strength. The obtained strength was compared to the baseline strengths obtained in the mechanical test section (figures 4 and 5).

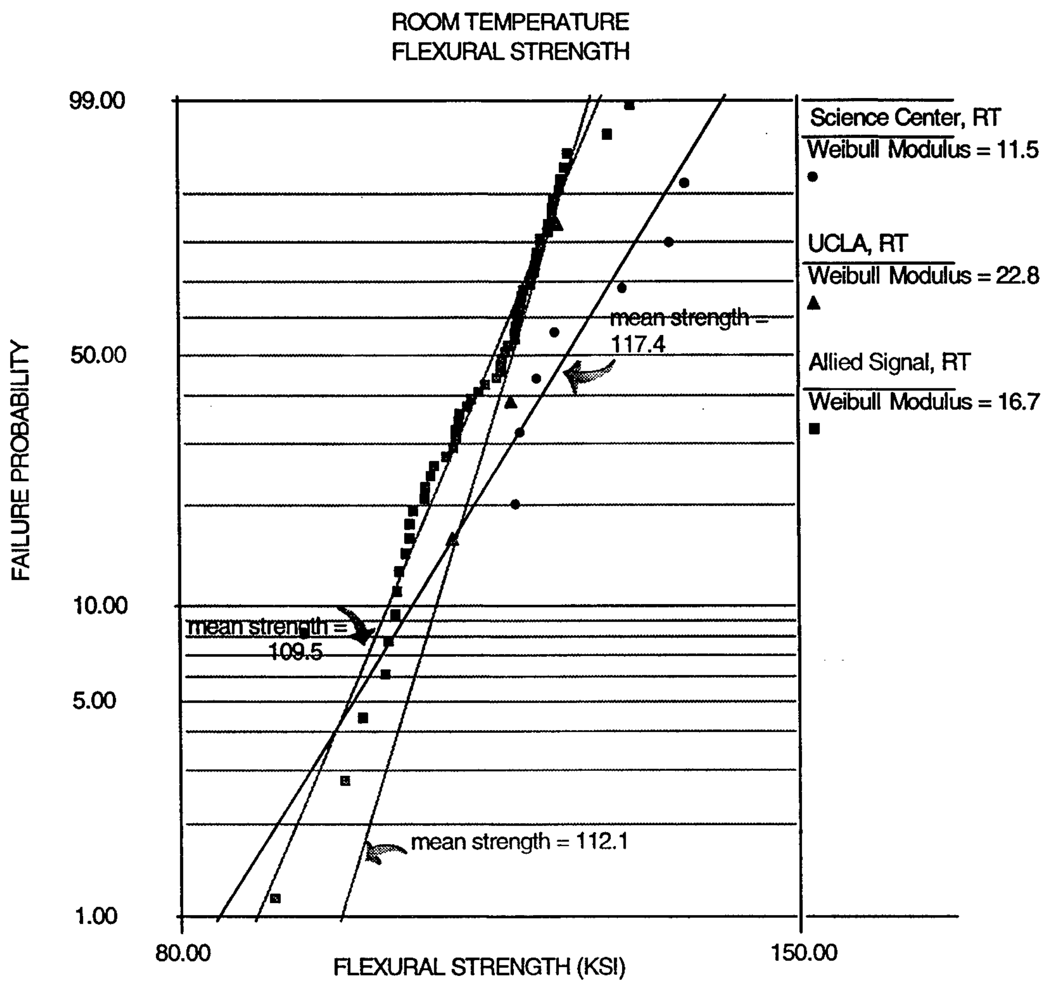


Figure 3- AS800 Silicon Nitride Flexural Strength Weibull Moduli Comparisons

INTERRUPTED STRESS RUPTURE

The purpose of the interrupted stress rupture tests is to establish the stress level below which AS800 is capable of sustaining a specified load for a specified amount of time. In the case of testing for LWTCA the specified time was set at 125% of the design life for the chamber calculated to be 33 hours and 20 minutes. The specified stress was set at 110% of the design stress. Design stress was taken

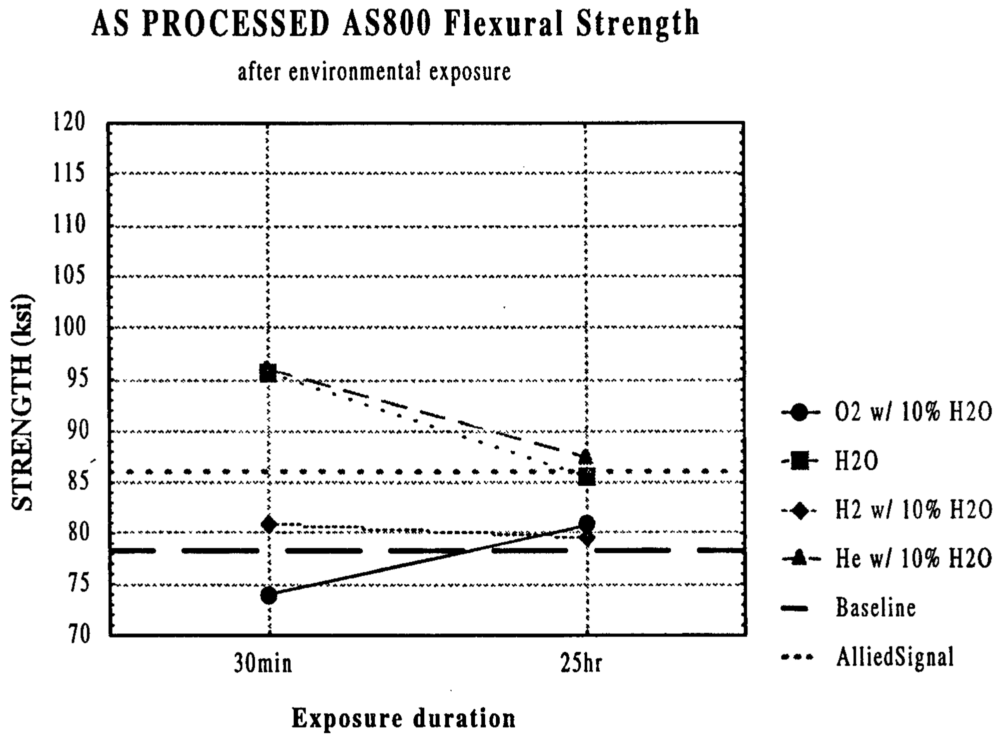


Figure 4 - As-processed AS800 flexural strength after environmental exposure

at the 90/95 limit strength calculated for AS800 based on the tensile data and statistical manipulations (Table II). Of the six specimens only one specimen survived the 33 hour and 20 minutes. The rest failed at the times summarized in Table III.

Due to these early failures, the applied stress level was revisited and reduced to 100% of the design stress for a repeat set of tests. Six more specimens were tested at the revisited applied stress level (Table II). Five of the specimens survived that entire duration of the applied stress and resulted in fast fracture strengths as summarized in Table IV.

THERMAL SHOCK

Thermal shock tests for 50 cycles from 1900°F to LN_2 quench followed by 50 cycles from 2200°F to LN_2 quench were performed on a test specimen 1"x 2"x 1/4". Data from this experiment suggests that there may be some accumulated damage during severe thermal shock. However, the population is too small to quantify the effect.

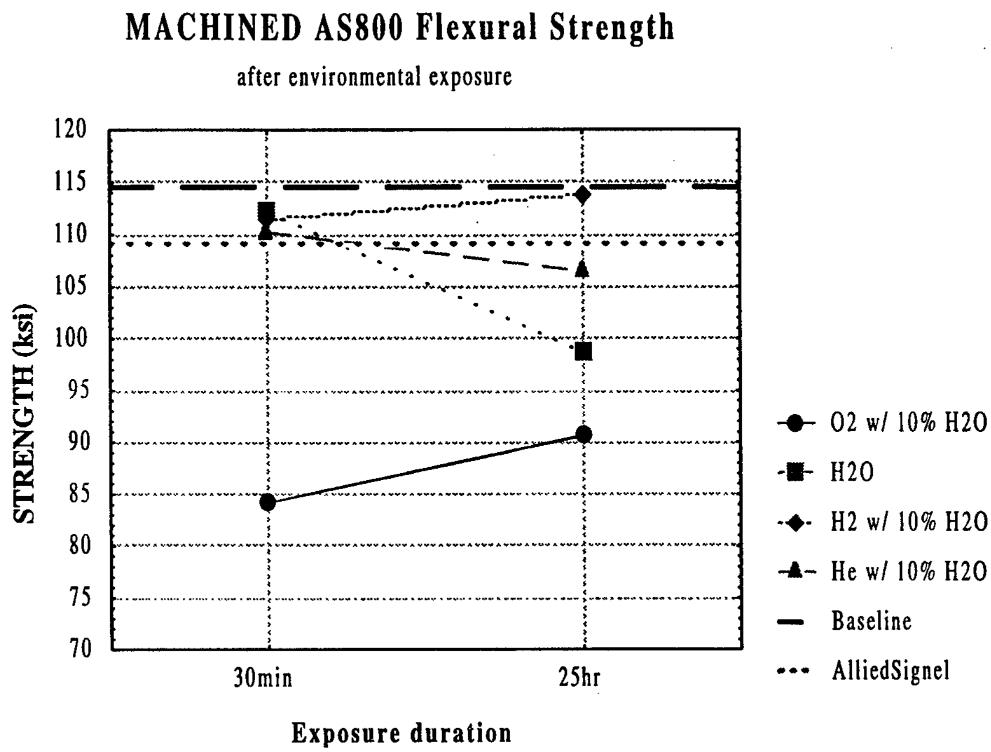


Figure 5 - Machined AS800 flexural strength after environmental exposure

Table II– Original and revised stress levels for AS800 interrupted stress rupture

Test Temperature	Applied Stress	Revised Applied Stress
-320°F	88 ksi	80 ksi
Room Temperature	84 ksi	76 ksi
1000°F	75 ksi	68 ksi

Table III – Failure times for AS800 specimen under 84 ksi sustained stress

Sample	Failure Time	Retained Strength (ksi)
AS24-RT1	9 hours, 3 min	
AS24-RT2	27 minutes	
AS24-RT1	33 hours, 22 min	94
AS24-RT1	< 1 min	
AS24-RT1	23 hours, 6 min	
AS24-RT1	1 min	

Table IV - AS800 retained strength after interrupted stress rupture, hold at 68 ksi for 33.3 hours

Test Temperature	Ultimate Strength (ksi)
-320°F	100.6
Room Temperature	89.9
1000°F	78.9

Average retained strength of these six tests was 84.9 ksi with a standard deviation

Another thermal shock test for a total of 100 cycles from 2200°F to LN_2 quench was also performed on a specimen 1"x 2" x 1/4". After the thermal shock test, the specimen was sectioned into 4 specimens and the specimens were placed with their LN_2 quenched surface face-down in the four-point bend test rig. The average retained strength of these four tests was 103.5 ksi with a standard deviation of 10.7 ksi. This data is consistent with the baseline data and, in itself, suggests that thermal shock has no detrimental effect on the AS800.

INDENTATION STRENGTH FRACTURE TOUGHNESS

To measure fracture toughness inexpensively and indirectly, four-point bend specimen were indented by a 20 Kg Vickers hardness tester and then tested in a 4-point flexural setup by UCLA. A typical indentation on the surface of an AS800 specimen is shown in figure 6. In addition to yielding fracture toughness data for AS800, two other objectives were met by this testing: the retained strength of AS800 was measured after a known flaw was introduced, and effects of cryogenic and high temperatures were evaluated on specimens with the known flaws. The test results were then compared to unindented specimens as baseline and correlated via a table relating indentation size to K_{IC} numbers.

Table V -Fracture Toughness via Vickers indent (20Kg) followed by standard four-point bend testing

Four-Point Bend Test Temperature	Flexural Strength		K_{IC}	
	Average	Standard Deviation	Average	Standard Deviation
-320°F:	55.7 ksi	1.6 ksi	8.7 ksi $in^{1/2}$	0.17 ksi $in^{1/2}$
Room temperature:	50.0 ksi	0.78 ksi	8.1 ksi $in^{1/2}$	0.12 ksi $in^{1/2}$
1832°F:	54.8 ksi	3.3 ksi	8.6 ksi $in^{1/2}$	0.35 ksi $in^{1/2}$

In summary, the 4-point bend results for AS800 at room temperature showed a mean strength of 112.1 ksi, compared to AlliedSignal's published data of 109.45 ksi. The results showed a 54% drop in 4-point bend strength after a 20Kg Vicker's indentation, which is consistent with AlliedSignal's results (Table V). A Weibull graph (figure 7) summarizes all the test results and shows that the family of data generated by Rocketdyne falls within data published by AlliedSignal.

The fracture toughness test results show that when a known flaw in introduced, the sample strength falls about 50% (as expected), and that the K_{IC} numbers are in the range of 9 MPa $m^{1/2}$ (8.2 ksi in $^{1/2}$).

COMPACT TENSION SPECIMEN TESTING

The objective of this test was to determine the fracture toughness of AS800 via industry-accepted Compact Tension testing. ASTM E399 was used as a generic procedure. The notable exception to the ASTM procedure was the shorter than normal initial crack length and the crack geometry.

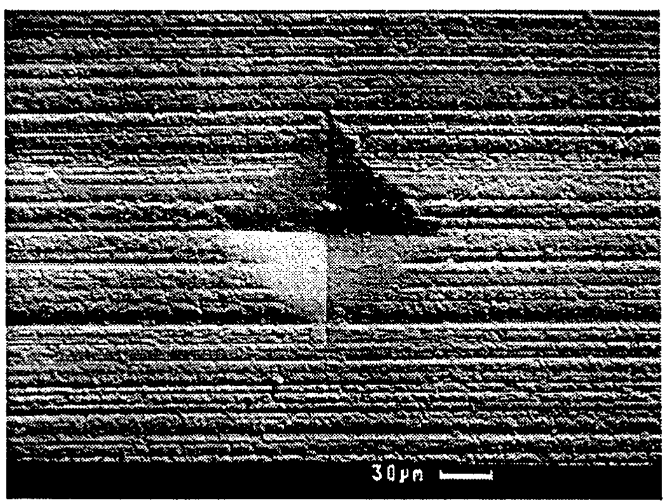


Figure 6 - Vicker's indentation in AS800 specimen

Figure 8 shows the Compact Tension specimen that was fabricated for this testing, the notch geometry is visible in this picture. Due to the non-plasticity of the AS800 material, it was thought that a fine crack was sufficient to initiate the fracture.

An attempt was made to follow ASTM E399, but since the material is ceramic and E399 is applicable to metallic materials, some experimental "interpretation" was necessitated. The results are summarized in Table VI.

THERMOMECHANICAL FATIGUE

The purpose of the thermomechanical fatigue tests was to determine if any property degradation would occur in the AS800 silicon nitride after exposure to

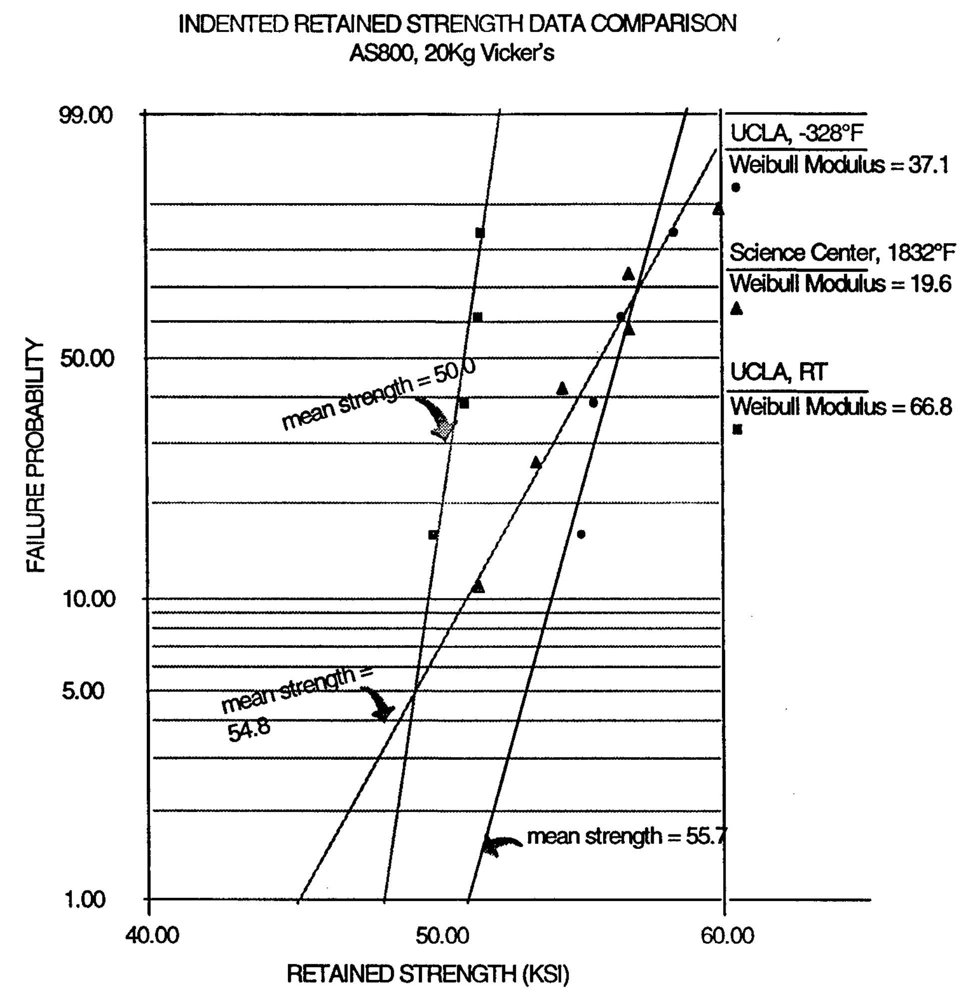


Figure 7 -AS800 retained strength data with known flaw

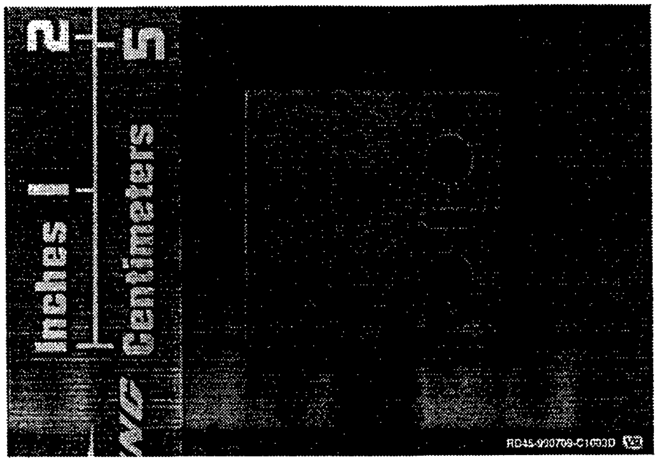


Figure 8 - Compact tension fracture toughness specimen

Table III - AS800 compact tension fracture toughness (Modified ASTM E399)

Test Temperature	Normal Statistics		Weibull Statistics	
	Average	Standard Deviation	Characteristic Strength	Weibull Modulus
-320°F:	7.8 ksi $in^{1/2}$	0.32 ksi $in^{1/2}$	7.1 ksi $in^{1/2}$	41.0
Room:	7.3 ksi $in^{1/2}$	n/a	n/a	n/a
1000°F:	7.0 ksi $in^{1/2}$	0.32 ksi $in^{1/2}$	n/a	n/a

thermal cycling typical for the LWTCA engine hardware under applied stress. Four Science Center hour-glass specimens were tested for 300 cycles followed by a tensile testing. No property degradation was observed.

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